CHEMICO MECHANICAL STUDY OF PORTLAND LIMESTONE CEMENT (PLC) DEVELOPED IN INDIAN SCENARIO

SHORT SYNOPSIS

Submitted in partial fulfillment of the requirement of the degree of

DOCTOR OF PHILOSOPHY

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by

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NAAC ACCREDITED 'A' GRADE INSTITUTION)
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DECLARATION BY THE SCHOLAR

I hereby declare that the short synopsis "Title **CHEMICO MECHANICAL STUDY OF PORTLAND LIMESTONE CEMENT (PLC) DEVELOPED IN INDIAN SCENARIO**" being submitted in partial fulfillment of the requirement for the degree of Doctor of Philosophy in department of **CHEMISTRY** under Faculty of **APPLIED SCIENCES** of Manav Rachna University, Faridabad during the academic year 2019-20.

It is a bona-fide record of original work carried out under the supervision of **Dr. Megha Bansal** and **Dr. B.N. Mohapatra** has not been submitted in full or part to any other university or institute for degree or diploma. Further, it is certified that short synopsis is within the plagiarism limit and copy of the same is attached as per university norms.

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ABSTRACT

This research proposal is aimed to develop the Portland Limestone cement in Indian Scenario. In the world, India is the second-largest producer of cement after China. The current annual installed capacity of cement industry in India in 2018-19 is 537 million tonne with cement production of around 337 million tonne. Housing & real estate, public infrastructure and industrial development are the three major sectors in India which demands huge supply of cement. The cement production is estimated to touch 550 MT by 2020. In the process of cement production especially during calcinations, **0.67 tonne of CO_2 gas is emitted during 1 tonne of cement production in 2017**. It is established that more the clinker factor, more will be the CO_2 emission. If we reduce the clinker factor, we can directly reduce the Green House Gas (GHG) emission.

In many countries like Europe, U.S., Mexico, Germany, New Zealand, China etc. the Portland limestone cement (PLC) is being manufactured by inter-grinding/inter blending the limestone (6-35%) with Portland clinker and gypsum. In India, the limestone is blended only up to 5% as a performance Improver. If the percentage of limestone is increased gradually, we may reduce the clinker factor and ultimately curb GHG emission to a greater extent. The availability of limestone is not the big issue for cement plants as limestone is the primary raw material for clinker production. Thus, the major benefits to develop PLC are a) Reduction in GHG emission, b) reduction of clinker factor c) reduction in fuel consumption.

To address the ever-increasing demand of cement, development of PLC in Indian scenario may come out as the best option.

Key words: Portland limestone cement, GHG emissions, clinker factor, interblending, performance improver.

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Introduction

The word "Cement" derived from Roman term opus caementicium. It is basically inorganic compound and acts as a binder to form concrete when bind with any aggregate in the presence of water. Cement is the combination of clinker and gypsum ($CaSO_4.2H_2O$). Clinker is formed by calcinations of limestone and clay.

Presently in India BIS has approved 14 types of cement to be manufactured in India like, Ordinary Portland cement (33, 43, 53 grade), Portland slag cement, Portland pozzolana cement (fly ash and calcined clay based), white Portland cement, high alumina cement, Super sulphated Cement, Rapid Hardening Portland cement, Masonry cement, oil well cement, Sulphate resisting Portland cement, Hydrophobic Portland cement, low heat Portland cement and composite cement. In India, mostly OPC (Ordinary Portland Cement), PSC (Portland Slag Cement) & PPC (Pozzolana Portland Cement) are being used for construction purpose. The current annual installed capacity of cement industry in India in 2018-19 is 537 million tonne, with cement production of around 337 million tonne.

Indian cement industry has been working on the issue of greenhouse gas emissions and reduced the CO_2 emission factor from 1.12 tonne of CO_2/tonne of cement in 1996 to 0.719 t of CO_2/t of cement in 2010. Moreover, the CO_2 emission factor has further reduced by 6.8% to 0.57 t of CO_2/t of cement in 2017. CO_2 emissions are targeted to be further reduced to 0.35 t of CO_2/t of cement by 2050.

The share of blended cement in the total quality of cement manufactured in India in 2010 was 68% and has increased to 73% in 2017.

Blended cement such as PPC, PSC and Composite cement have a key role in the reduction of the clinker factor. The clinker factor improved from 0.74 in year 2010 to 0.71 in the year 2017.

There are concerns for the sustainable development and environmental impact in the cement and building material industry. To further reduce the energy consumption and CO_2 emissions, the production of Portland Limestone Cement (PLC) is described as an economical and environmental friendly solution for cement industries around the world.

PLC (Portland Limestone Cement) is prepared by inter-grinding of clinker, limestone and gypsum. PLC used in various parts of world mainly Europe (Portugal, The Netherlands, Germany, France, Finland, Austria, Spain, Sweden & United Kingdom) and America.

According to European cements standard EN 197-1 (BS-EN197-1, 2000) Cement-Part 1 the CEM Portland cement was categorized into following groups:

Sl. No.	Category	% of Clinker
1	CEM I Portland cement	>95
2	CEM II Portland-composite cement	65-94
3	CEM III Blast furnace cement	5-64
4	CEM IV Pozzolanic cement	45-89
5	CEM V Composite cement	20-64

Around 8 per cent of global CO_2 emissions are coming from cement industry. Worldwide, cement industry is facing challenges in reducing CO_2 emissions and conserving the raw material with energy resources. The main aim of cement producers is to increase energy efficiency and the use of AFR (Alternative Fuel and Raw Materials). In cement plant, more than 50 % of the CO_2 emitted during calcination of limestone, 40 % due to combustion of fuels in the kiln and 10 % from other cement plant operations (Imbabi et al., 2012)

Many studies revealed that inter-grinding of clinker with different proportion of limestone resulted in improved grinding behavior of clinker which directly save the electric cost, decrease in fuel consumption and grinding time, many researchers showed that hydration rate of primary phase of clinker, i.e., alite (C_3S) is increased when the amount and fineness of limestone is increased. Workability of mortar paste can also be improved by using cement containing limestone due to its particle size distribution; cement industries release Green House Gas (GHG) with different gases to the atmosphere, NO_x, Dioxin, SO_x, and CO_2. Out of many ways for reducing GHG emission, blending or inter-grinding limestone with Portland cement is one of the techniques, for reducing the CO_2 emissions, climate change, economic and technical benefits (Damtoft et al., 2008).

In near future the scarcity of cement grade limestone availability is biggest challenges on the other hand there is huge availability of marginal or low-grade limestone and dolomitic limestone deposits in India. If we are in the position to use marginal or low grade and dolomitic limestone in

cement production just like other countries i.e. European and America, it would be the biggest achievement for Indian Cement industries. The answer is Portland limestone Cement (PLC).

This research proposal is first of its kind to propose synthesis of PLC in Indian Scenario using various qualities of clinker and limestone. The raw materials will be taken from all major sites of India covering North, South, East and West so that a clear picture is obtained. The aim of proposal is to develop a stable formulation / composite for PLC.

Literature Review

This section of research proposal provides an overview of past research in related topics covered by this work.

Manufacturing: Cement is originated from is ancient Rome & Greece. A hard mass was formed when volcanic ash and lime reacted in the presence of water (Baron and Douvre, 1987).

Joseph Aspdin, a bricklayer and mason in Leeds was granted a patent in 1824 for material that was invented by mixing and heating the limestone with clay. He called it "Portland cement" due to its resemblance to the color of stone found on the Isle of Portland. By the early 21st century, World leaders in cement production are China and India, presently China and India producing 220 million metric ton per annum and 337MT per annum of cement respectively. USA, Russia, Vietnam, brazil, turkey, Iran and Indonesia are the major cement producing countries after China and India. The Composition of Portland cement is

Portland cement = lime (CaO) + silica (SiO_2) + alumina (Al_2O_3) + Iron oxide (Fe_2O_3). Lime is obtained from calcareous material while silicon di oxide, alumina and Iron oxide are derived from argillaceous material. Other necessary raw material is gypsum$CaSO_4$. $2H_2O$, which is acts a set retarder, around 5 percent of gypsum is added (Bonavetti et al., 2003). Manufacturing processes of Portland cement has four stages: (a) crushing and grinding (b) blending (c) burning and (d) grinding of final burned product. The material obtained after burning is known as clinker which contains 5 % of gypsum. Cement manufacturing processes are of three types wet, dry, and semidry however, most commonly used is dry process. For grinding of the raw material to a fine powder generally, ball and roller grinding mills are used. This ground raw mix powder for further homogenisation goes to storage point called as raw meal silos.

The blending process will affect the cement quality (Barrett et al., 2013). Quality of raw material and Portland clinkers depend on Lime Saturation Factor (LSF), Silica Modulus (SM), and alumina Modulus (AM). LSF, SM, and AM can be determined by the oxide of lime, silica, alumina and iron which are presented in cement raw material (Bensted, 1980). In the process of clinker manufacturing the raw mix (blended mixture of crushed raw material) is heated and burned at very high temperature in the kiln. During this process the raw material is converted into granular material termed as clinker. This process of clinker formation takes place at very high temperature

in Rotary Kiln which rotates between 30 and 250 revolutions per hour. To generate heat in the Rotary Kiln, pulverized coal and coke or other fuel is burn at the bottom end of kiln. Ash of injected fuel plays a very important role because it combines with raw mix (Bosoaga et al., 2009). End product of the kiln is Clinker nodules of various shapes and sizes. These hot clinkers nodules enter into coolers, for cool down the hot clinker for better phase formation. As per requirement, the clinker may be stored or ground to cement **(**Campiteli and Florinda, 1990**).** The cooled clinker is then stored into clinker silos; the required amount of gypsum is added to form cement.

Structural properties: The strength given by any Portland cement depends on granulometry, morphology and phase composition. The early strength is mainly given by alite and ultimate strength is depends on belite. Contribution to strength by celite and ferrite is very little. On hydration C_3A produces high heat of hydration followed by C_3S, C_4AF, and C_2S. American Society for Testing and Materials **(ASTM)** standardized five types of Portland cement in the United States: Type-I (Ordinary), Type-II (Modified), Type-III (High-early-strength), Type-IV (Low-heat) & Type-V (Sulfate-resistant).

Masonry cement is specially used for plastering work, where strength is not required. Oil well cement is use when there is requirement of cementing work in the drilling of oil wells, where they are subject to high temperatures and pressures.

Development of cement in Indian Scenario: In Madras (1904) the first cement factory of India was established. And then, in 1912-13, three cement factories were established at Katni (M.P.), Porbandar (Gujarat) and Bundi (Rajasthan). The cement production was increased from 945 in 1914 to 84,300 tonnes in 1918. In 1926, the Indian cement manufacturers' association was formed. In 1936, ACC (Associated cement companies limited) was formed by the merger of 10 principal cement companies. In 1947, at the time of partitioned in 1947, the total capacity was 2.2 MT with 23 cement plants. As on April 30, 2003, these were 124 large cement plants (with capacity of 140.02 MT).

Portland cement is manufactured by inter-grinding of clinker with 5% of gypsum. Approximately >50% of CO_2 emitted during calcination (decarbonation) of limestone, 40% by combustion of fuel, and rest 10% of CO_2 emissions during the process of quarrying and transportation of products (Bosoaga et al., 2009). Clinkerisation is the process, when at 1450 ◦C lime (CaO) and silicon

dioxide SiO_2 are combined in rotary kiln. Four major clinker phases like: Alite, Belite, Celite and ferrite are formed during this process of clinkerisation (Imbabi et al., 2012).

PLC is an upgraded version of **Portland cement** which has improved environmental footprint and better performance of concrete. PLC contains between 6% to 35% **limestone.** Clinker content is directly reduced by increasing the percentage of limestone in Portland cement. PLC is manufactured by inter grinding Portland clinker with 6-35% limestone, but quality of clinker should be is same as that of OPC. Limestone quality is responsible for the performance of PLC. High quality 6-35% limestone ($CaCO3 \geq 75\%$) replaces Portland clinker in the making of Portland limestone cement (PLC). **Development of strength in PLC is due to the formation of hemi and mono carboaluminate.** The limestone physically densifies the micro structure of concrete due to its filler effect. Limestone is ground more finely than regular Portland cement, because limestone softer than traditional clinker. Mohs' hardness of limestone is less than traditional clinker. Strength of the resulting concrete using PLC depends on distribution of the particles. These properties of PLC give particle packing effect, which increase the resulting strength of the concrete. Previously researchers believed the limestone serves as inert filler, but many researches showed that limestone can react to some extent. As the fineness of limestone's particles increases, it will enhance the reaction (Soroka and Setter, 1977). Limestone reacts completely to form different phases of carboaluminate (Matschei et al., 2007).

Development of Portland limestone cement in Global Scenario: Germany was the first country to blend 20 % limestone in cement in 1965 then after in 1979 French cement standards also allowed addition of limestone addition followed by Canadian standard Association in 1983. In 1992, UK specification permitted up to 20% limestone addition in cement. In 2000, European Committee for Standardization adopted EN 197-1 specifications which allowed 5% blending of limestone in all 27 types of cement. Limestone is used at higher levels in CEM II cements ranging from 6-35 %. Clay content in limestone should be Max 1.2g/100g which can be determined by MBI (Methylene Blue Index). **Limestone as ingredient in cement industry is also uses in several other** countries outside of Europe (Ewais et al., 2009). Around 39 listed countries allowed mineral additions from 6 to 20% as per *Cement Standards of the World* (Pandey and Sharma, 2002). But out of above countries, both former USSR and China permitted up to 10% limestone.

One of the greatest advantage of blending limestone is that it will reduce the clinker content. Directly it reduces both the energy consumption and CO_2 emissions (and other greenhouse gases). In cement plant from two sources carbon dioxide emitted: a) Limestone calcination, and b) burning of fuel. Production of other blended cements (PPC & PSC) although will reduce both the clinker content and GHG (CO_2), on the other hand while using of limestone as a blending component would give another option to improve sustainability characteristics (Matschei et al., 2007; Phair, 2006).

Producing PLC reduces up to 10 % Green House Gas (CO_2 emissions) than that of manufacturing conventional Portland cement by blending limestone. Producing PLC will directly conserve the fuel and high-grade limestone however it has also been reported that increasing percentage of limestone will decrease water requirement. (Tsivilis et al., 1999; 2002).

Description of Broad Area

Following reactions occurred during the formation of Portland clinker: the first stage is calcination process in which limestone is heated to around 1450ºC. In this reaction large amount of CO_2 is emitted and lime is produced. The produced lime combines with argillaceous raw materials (source of silica, iron oxide and alumina) and form final product 'CLINKER' in Rotary Kiln. Major source of CO_2 emission in the manufacture of Portland Clinker is calcination process and other source is burning of fuel. Final product generated from rotary kiln is Clinker (a small dark nodule). In India there are generally 14 types of cement used on the bases of their unique properties, Types of Portland cement in world: Table -1 show the types of cement manufactured in India.

Table 1: Specification of cement as per Indian Standards

Sl. No	Indian Standard No.	Ref. Yr.	Amend ments	Title of Indian Standard
1	IS 269:2015	-	-	Ordinary Portland Cement
2	IS 455:2015	-	-	Portland Slag Cement
3	IS 1489 (Part 1):2015	-	1	Portland Pozzolana cement (Fly ash based)
4	IS 1489 (Part 2):2015	-	-	Portland Pozzolana cement (Calcined clay based)
5	IS 3466:1988	2013	5	Masonry cement
6	IS 6452:1989	2019	5	High alumina cement (Structural)
7	IS 6909:1990	2016	5	Super Sulphated Cement
8	IS 8041:1990	2019	4	Rapid hardening Portland cement
9	IS 8042:2015	-	-	White Portland cement
10	IS 8043:2019	2019	3	Hydrophobic Portland cement
11	IS 8229:1986	2013	6	Oil-well cement
12	IS 12330:1988	2019	6	Sulphate resisting Portland cement
13	IS 12600:1989	2019	5	Low heat Portland cement
14	IS 16415:2015	-	-	Composite cement

In America ASTM (American Society for Testing and Materials) standardized five types of Portland cements:

Type-I Ordinary
Type-II Modified
Type-III High-early-strength
Type-IV low-heat
Type-V Sulfate-resistant

As per ASTM C-595 cement are four types:

Type IL - Portland limestone cement
Type IS - Portland slag cement
Type IP - Portland Pozzolana cement
Type IT- Ternary blended cement

Portland Limestone cement is advanced form of Portland cement which reduces environmental carbon footprint significantly improve concrete performance. The usage of PLC increased tremendously in last decades due to various factors such as reduced consumption of raw materials and less utilization of energy during clinker production (Damtoft et al., 2008; Kenai et al., 2004; Thomas et al., 2010; 2013). Blending of limestone also affects Blaine values and it is desired that post limestone blending the grinding should be highly effective to get desired strength and high Blaine values (Voglis et al., 2005).

CO2 emission will rise from 2 billion tonnes per year in 2010 to 4.8 billion tonnes per year in 2050. Limestone is easily available in cement plant for production of PLC, as this is a primary raw material for clinker production, so in this way transport cost will also not increase. Since 1990 there is 4-fold increase in the use of Portland limestone cement in Europe instead of use of PPC and PSC (Tennis et al., 2011). Use of limestone will reduce the clinker content and directly reducing the greenhouse emissions (CO2), higher contents of limestone is continuing use for further development. Table-2 below reflecting the results of energy and emissions reductions annually while using different dosage of limestone in making of PLC.

Table-2: Energy usage and Emissions (Annual Reduction)

	10% Limestone*	**15 % Limestone***
Fuel (million BTU)	4,43,00	6,64,000
Electricity (kWh)	6,970,000	10,440,000
Emissions Reduction		
SO2(lbs)	5, 81, 000	8,70,000
NOx (lbs)	5,80,000	8,70,000
CO (lbs)	1,04,000	1,55,000
CO_2 (lbs)	1,89,000	2,83,000
Total Hydrocarbons, THC (lbs)	14,300	21,400

*** Per million tons of cement**

Objectives of the Study:

1. Chemical analysis of various raw materials i.e., clinker, limestone and gypsum obtained from different zones of India.
2. Grindability Index of different type of Clinker and limestone of variable grades (Low, Cement & High grade) samples.
3. DTA/TGA analysis of various limestone samples.
4. Mineralogical analysis and Petrographic analysis of various limestone and clinker samples by XRD and OM respectively.
5. Preparation of Portland Limestone Cement (PLC) using different dosages of limestone (different qualities of limestone with variable quantity of $CaCO_3$ and $MgCO_3$) and fixed percentages of gypsum.
6. Physical and chemical characterization of synthesized PLC.
7. Performance characteristics of PLC using DTA/TGA, calorimetry, XRD, Particle size analyzer and optical microscopy technique.

Methodology to be adopted:

A) Collection of raw materials (limestone, clinker and gypsum) from four cement plants located at all four zones of India, i.e. North, south, east and west, so that, we can optimize the PLC in the better way.

B) Sample preparation: crushing for size reduction, quartering and coning

C) Materials characterization: chemical, physical, mineralogical, morphology and granulometry of the all collected materials.

i) Chemical analysis of clinker, limestone and gypsum samples by as per IS: 4032-1985, IS: 1760(Part-1, 2, 3, 5 & 6), IS: 1288-1985 and NCB standard methods.

ii) Bond Index of Grindability Index of all collected limestone samples to check grinding energy required to crush as per Indian standard IS 10218: 1982 (Method of determination of grindability Index).

iii) Thermal analysis by DTA and TGA (PerkinElmer Model-Diamond TG/DTA).

iv) Mineralogical estimation of raw materials by XRD (X-ray Diffractometer), Make Rigaku International Japan, Model-Smart Lab

v) Microscopic analysis of clinker samples by Optical microscopy Make: Nikon Japan, Make-POL 100, clinker samples prepared for microscopic analysis with the help of grinding and polishing machine.

f) Petrographic examination of limestone samples by Optical Microscope; Make: Nikon Japan, Make-POL 100.

D) Preparation of Portland limestone Cement (PLC) blends (6Kgs each) by intergrinding of clinker, limestone and gypsum to the fineness ($380\pm20 m^2/Kg$). like in table below, means in A-CTL(control sample i.e, blends prepared without taking limestone), A-PLC-5(PLC blends prepared by taking limestone up to 5%), A-PLC-10(prepared by taking limestone up to 10%), A-PLC-15(limestone up to 15%), A-PLC-20(limestone up to 20%), in all below PLC blends we have to maintain the SO_3 % up to (3%).

Naming of PLC blends	% of Clinker	% of Limestone (different grades of limestone like; LG/CG/HG)	% of Gypsum
A-CTL(N, E, W & S)	95	-	5
A-PLC-5 (N, E, W & S)	90	5	5
A-PLC-10 (N, E, W & S)	85	10	5
A-PLC-15 (N, E, W & S)	80	15	5
A-PLC-20 (N, E, W & S)	75	20	5

LG-Low Grade limestone, CG-cement grade limestone, HG- high grade limestone

E) Analysis of PLC blends:

i) Chemical analysis of different PLC blends.

i) Physical analysis of above blends as per IS: 4031; Normal consistency, specific gravity, fineness, setting time (IST & FST), Soundness, compressive strength and drying shrinkage.

ii) Distribution of Particle size using Particle Size Analyser (Microtrac Model: S3500)

Proposed/expected outcome of the research:

1. Proposing the optimized conditions for the production of Portland limestone cement in Indian conditions.
2. Limiting the use of energy and natural resources.
3. Incorporation of low and marginal limestone in cement manufacturing industries in various ratios.
4. Production of PLCs can also lead to an extended life of limestone quarries.
5. Use of PLCs containing limestone has the potential to reduce CO_2 emissions significantly.

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ABSTRACT

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Researcher : SANDEEP KUMAR GUPTA

Registration Number: 2K19 PFS 002

Supervisor: Dr. MEGHA BANSAL

Department: Chemistry

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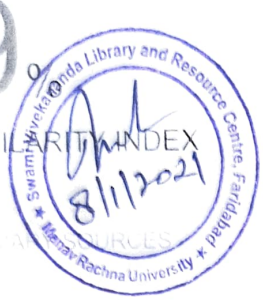

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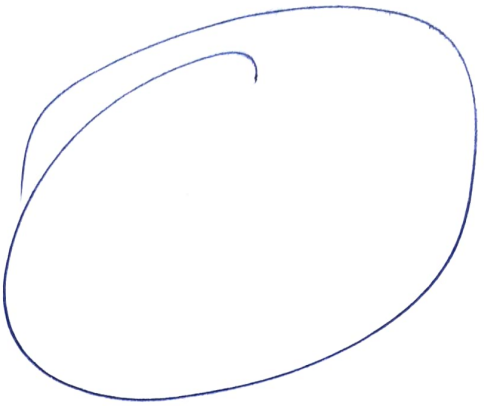
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